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Plasma-Surface Interaction and Processing of Materials
NATO Advanced Study Institute, Alicante, Spain, September 4-16, 1988

ASI Directors:

Prof. Orlando Auciello
N.C. State Univ.
Dept. Nucl. Eng.
Raleigh, NC 27695-7909, USA
TE: (919) 737-7662
Telex: 9109974599 MEDIARALEIGH

AD-A213 602

Prof. Alberto Gras-Marti
LCFCA, Universitat d'Alacant
E-03080, Alicante, Spain
TE: (96) 566-1150 Ext. 1165
Telex: 666-16 UDEAE

Dr. Daniel L. Flamm
AT&T Bell Laboratories
Murray Hill, NJ 07974, USA
TE: (201) 582-5306
Telex: 13-8650

Advisory Committee:

Prof. Hans H. Andersen
Physics Lab. II
H.C. Orsted Inst.
Universitetsparken 5
DK-2100 Copenhagen O
Denmark
TE: 45-1353133

BOOK OF ABSTRACTS

Prof. Dave G. Armour
Univ. of Salford
Dept. Electr. Eng.
Salford M54WT
United Kingdom
Telex: 668680 (Sulib)

Prof. Ricardo D'Agostino
Univ. Degli Studi Bari
Dept. di Chimica
173 Via Amendola
70126 Bari, Italy
Telex: 812274 CHIMIC I

Prof. A. Ricard
Univ. de Paris-Sud
Lab. de Physique des Gaz
et des Plasmas
91405 Orsay-Cedex, France
TE: 941.72.51



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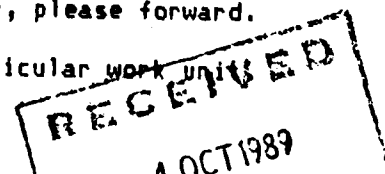
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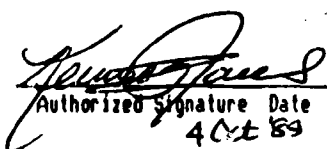
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BASIC PHYSICS OF PLASMAS/DISCHARGES

A. Ricard

L.P.G.P. - Bâtiment 212

Universite Paris-Sud - 91405, Orsay Cedex - France

Introduction

The scope of this lecture is to analyze the production of active species in low pressure gas discharges for surface treatments. Electron collisions are the main source of active species in the discharge volume. The physics of electron excitations will be described and applications to rare gases (He, Ar), diatomic molecules (H_2, N_2O_2) and an electronegative gas such as CF_4 will be considered.

Production of nitrogen excited species in flowing discharges and post-discharges will be specially studied. Mixtures with reactive gases are used for surface treatments as Ar - N_2, O_2 , $N_2 - O_2$, $CF_4 - O_2$ and so on.

Excitation transfers between active species are relevant phenomena. They will be treated in the cases of He, Ar, N_2O_2 metastable species reacting on molecular gases.

SOME PLASMA/DISCHARGE DEFINITIONS

D.C., R.F. and microwave glow discharges will be briefly described. Characteristics of low pressure, out of equilibrium, plasmas will be given such as ionization degree, electron and neutral temperatures.

ACTIVE PLASMAS

The production of active species will be studied by considering radiative and metastable states, atoms and radicals issuing from molecules. A balance between excitation by electron collisions and losses by diffusion on the walls, neutral and electron collisions will be established. Cases of Ar, N_2, O_2 and CF_4 will be analyzed. The production of the nitrogen active species in flowing discharges and post-discharges will be studied by focusing oneself on long living species which are not destroyed on the reactor walls.

REACTIVE PLASMAS

The reactions between metastable atoms or molecules and other molecular species will be analyzed by reviewing Penning ionizations and excitation transfers. A comparison between metastable transfers and ion charge exchanges will be given with applications to collisions of He, Ar, N₂, O₂ metastable on molecular gases as H₂, N₂, O₂, CH₄, NH₃, NO and so on.

CONCLUSION

Some correlations between plasma excited state production and film characteristics will be presented for He - Si H₄ PECVD, metal surface nitriding in N₂ post-discharges and polymerization in CF₄ plasmas.

LOW TEMPERATURE PLASMA CHEMISTRY

Daniel L. Flamm
AT&T Bell Laboratories
600 Mountain Avenue
Murray Hill, NJ 07974

Plasma etching and chemical vapor deposition offer low temperature processing, fine resolution, and adaptability to high throughput and automation for microcircuit fabrication. Plasma processes are also receiving increased attention for other applications such as surface treatment and diamond growth. This lecture introduces the plasma chemistry, physical variables, reaction and transport phenomena that underlie these applications.

Discharge properties are adjusted with operating variables, such as power, pressure, temperature, excitation frequency and reactor design. Relationships among these adjustable parameters and similarity variables will be discussed, along with their effects on active species production, the flux of reactants impinging on surfaces, electron and ion energy in the plasma and other aspects of process chemistry.

Production etching processes can be characterized by their rate, selectivity, directionality, uniformity and surface morphology. Ultimately, these attributes are governed by four fundamental kinds of gas-surface interaction: physical sputtering, chemical etching, ion "damage" induced etching and "sidewall" inhibitor ion assisted etching. The mechanisms and chemistry of these interactions will be elaborated with selected examples from laboratory experiments and practical etching feed mixtures.

In conventional CVD, high temperatures supply the activation energy for both gas phase and surface reactions, and thermally-driven rearrangements lend stability and inertness to deposited films. Carefully selected plasma chemistries and electron-energy driven reactions can partially compensate for a lower

gas temperature, while ion bombardment promotes low temperature surface reconstruction and radical species passivate dangling bonds. The plasma chemistry and discharge techniques used to control CVD processes are similar to those in plasma etching. Deposition of hydrogenated and fluorinated silicon nitrides will be examined as a case in point.

OPTICAL DIAGNOSTIC TECHNIQUES FOR LOW PRESSURE PLASMAS AND PLASMA PROCESSING

V. M. Donnelly
AT&T Bell Laboratories
Murray Hill, New Jersey 07974

Optical diagnostic techniques have greatly expanded our understanding of low pressure discharges used in etching and deposition processes for micro-electronics devices. This lecture will review and compare various techniques with particular emphasis on studies which are of relevance to low pressure plasma processing. Optical emission induced by electron-molecule collisions will be covered, with emphasis on refinements of the technique to provide more quantitative information. Absorption techniques will also be covered, and examples given of determinations of absolute number densities of stable molecules, as well as radicals. Laser spectroscopic techniques have rapidly evolved in recent years. The methods to be covered include laser-induced fluorescence, laser raman scattering, photogalvanic effects, and tunable IR laser absorption. Finally, surface diagnostic optical techniques will be briefly touched upon. Some of the methods have not yet been employed in plasma environments, but have the potential to yield valuable information.

MODERN PLASMA PROBE DIAGNOSTICS (EED Measurement)

Valery Godyak
GTE Products Corporation

The fundamentals of plasma probe diagnostics theory and technique will be given in the introductory part of the lecture. Basic formulae, requirements, and limitations for plasma probe diagnostics will be discussed. Emphasis will be placed on Electron Energy Distribution (EED) measurements in gas discharge plasmas. Two different approaches for EED measurements are considered. The first is the conventional method using a probe voltage modulated by a small rf signal with phase sensitive detection and integration. The second is the pulse method with boxcar averaging.

Different kinds of distortions in EED measurement due to probe-surface contamination, change in probe work function during the measurements, and plasma noise and oscillation will be considered. The system for absolute EED measurement which eliminates these distortions will be described. It is based on a combination of a fast pulse techniques, noise suppression feedback, and automated probe cleaning during the measurement.

It will be shown that state of the art analog and digital electronics allow EED measurements to be made in tens of μs , resulting in high energy resolution with a dynamic range of 30-50 db for single shot and 60-80 db for steady or repetitive processes.

As an illustration of the capability of the system for EED measurement, the results obtained in the positive column, cathode region and under conditions of artificial plasma perturbation will be presented.

In conclusion, some fundamental and technological limitations on speed, resolution and dynamic range of the fast EED measurement will be discussed.

TRANSPORT PHENOMENA IN PLASMA PROCESSING

J. A. Valles-Abarca
LCFCA, Universitate d'Alicant, Alicante, Spain

INTRODUCTION

- * Terminology
- * Significance of Transport Phenomena in Plasma Processing of Materials
- * Basic Concepts: Particle Interactions in a Low-pressure Discharge

TRANSPORT IN PLASMA PROCESSING: PHENOMENOLOGY

- * Charge
- * Mass
- * Energy
- * Momentum

TRANSPORT IN PLASMA PROCESSING: TREATMENTS

- * Analytical Treatments
- * Computer Simulations

CASE STUDIES

- * Electrode Bombardment in Glow Discharge Systems
- * Thickness Profiles in Sputter Deposition
- * Thermalization of Energetic Particle Fluxes
- * Pressure Effects in Gas Discharges

FUNDAMENTALS OF PHYSICAL SPUTTERING

H. Urbassek

Fundamentals of physical sputtering will be discussed.

BASIC PHENOMENA IN REACTIVE ETCHING OF MATERIALS

Orlando Auciello
North Carolina State University
Raleigh, NC 27695-7909, USA

Spontaneous and radiation-enhanced etching of materials due to their interaction with sub-eV chemically reactive species only and with the simultaneous action of radiation (energetic ions, electrons, photons), respectively, has become a subject of intensive research due to their importance in microelectronics, fusion and space technologies.

Existing evidence suggests that the basic processes that drive spontaneous etching of several etchant-material combinations may be similar: F/Si, H/Si, and H/C systems can be cited as examples. On the other hand, many etchant-material systems appear to involve system-specific processes that should not be generalized; examples are the XeF₂/Si and Cl/Si systems.

Experiments show that chemical etching, whenever this is the dominant phenomenon, is generally enhanced when the flux of thermalized active species landing on surfaces is accompanied by simultaneous energetic particle or photon bombardment. In etching of microelectronics materials, investigators have systematically studied etching product yield and product kinetic energy as a function of ion mass, ion energy and angle of incidence; data on temperature dependence of etching are more limited. By contrast, the temperature dependence of etching of C and C-composites in fusion technology has been extensively studied, but there is a lack of data on ion mass and incidence angle dependence, and on kinetic energy distribution of products. There appears to be no comparable data on radiation enhanced etching of materials used in the outer space environment surrounding the Earth. The fundamental parameters mentioned above in relation to chemical etching are essential for formulating and testing models of spontaneous and radiation-enhanced etching chemistry.

Spontaneous and radiation-enhanced etching may play a role also in thin film deposition. It has been observed, for example, that formation of diamond or diamond-like films can be greatly improved if simultaneous etching of a co-depositing graphitic phase is achieved.

Most recently, new selective etching techniques have been developed, which allows selective dry chemical etching of compound semiconductors, such as gallium arsenide phosphide. The technique exploits electronic differences in materials. Photons, impacting on the compound, carry a precisely determined amount of energy, sufficient to create free electrons and holes in the materials to be etched, but not in adjacent or underlying components of the compound semiconductor. This effect combined with the simultaneous exposure of the compound to reactive species produces the selective etching.

Etching of high temperature (T_c) superconducting materials is a new challenge presented to researchers. Particularly, the etching of high T_c films will be fundamental to the use of these new materials in manufacturing high T_c -based microcircuits for microelectronics applications.

Available data will be discussed in view of phenomenological models and more quantitative calculations recently developed to understand the complex phenomena involved in etching of materials.

THIN FILM DEPOSITION BY SPUTTERING

James M. E. Harper
IBM Thomas J. Watson Research Center
Yorktown Heights, NY 10598, USA

Deposition processes will be discussed in the following areas: (1) Sputtering fundamentals (yield, angular dependence; distribution of sputtered species); (2) Sputter deposition systems (DC, RF diode; Magnetron; Ion beam); (3) Multicomponent film deposition (alloys; compounds[reactive sputtering]). Also included will be discussions of properties of sputtered thin films, i.e., composition control; microstructure; step coverage; stress; effects of ion bombardment; effects of deposition temperature.

Examples will be taken mainly from the field of electronic materials for semiconductor technology (oxides, nitrides, aluminum alloys, refractory metals).

RBS, SIMS, AES AND ESCA ANALYSIS OF SURFACES

D. G. Armour

Department of Electronic and Electrical Engineering
University of Salford, Salford M5 4WT, U.K.

Ion beam and plasma assisted processes have been used to modify the mechanical, optical and electrical properties of surfaces and surface coatings. While the systems required to produce these property modifications are basically simple, the phenomena associated with the simultaneous deposition of energy and material are complex. In order to understand, reproduce or optimise the treatment conditions it is necessary to analyze both the composition and structure of the film or treated surface and to relate these parameters to the specific properties of interest.

Depending on the particular application, films of thickness ranging from a single monolayer up to several microns may be of interest. Despite this range, it has been found that the analytical techniques of most general value are those which possess high depth resolution. In principle, for thick coatings, profiling by removal of thin layers by sputtering (ion beam etching) or chemical etching enables the structure and composition of the coating to be investigated as a function of depth below the original surface using a high resolution analysis technique. This depth-resolved information is often more useful than an indication of the properties of the film averaged over the full thickness.

The present lecture, therefore, concentrates on high depth resolution, surface analytical techniques and their application to the analysis of surface coatings. This emphasis reflects the fact that many technologically important properties such as corrosion, adhesion and wear are controlled by the outermost surface layers. It is this fact, of course, that is responsible for the development of directed energy processes utilizing either plasmas or ion, electron

and laser beams to tailor the composition and structure of these layers. The techniques to be discussed are all based on the use of ions, electrons or photons as the probe and the measurement of the energy and intensity of the emitted radiation. In all cases, the energy of the emitted ion, electron or photon identifies the atomic species in the material and the yield indicates the amount of atoms.

Although basic analytical systems are conceptually simple, comprising a source, sample and detector arrangement, the demanding energy resolution, vacuum and data handling specifications means that their use requires a considerable capital investment. The high cost of analysis makes it essential to consider what information is required and which technique is most suitable for obtaining it. It is the purpose of the present lecture: (1) to review and compare the capabilities of some of the main techniques available; (2) to describe the physical basis of photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES), Rutherford backscattering (RBS), Ion scattering spectrometry (ISS) and Secondary ion mass spectrometry (SIMS); (3) to describe the main practical features of the apparatus required for these analytical techniques; (4) to illustrate the application of these techniques to the analysis of thin films.

SCANNING TUNNELING MICROSCOPY OF PLASMA-EXPOSED SURFACES

J. K. Gimzewski
IBM Research Division, Zurich Research Laboratory
CH-8803 Rschlikon, Switzerland

This talk concerns the application of the Scanning Tunneling Microscope (STM)[1] and related devices to local characterization of surfaces exposed to plasmas. An important feature of STM is its ability to image nonperiodic or disordered surfaces with high spatial resolution. In addition, the technique is a local probe enabling electronic structural and other information to be acquired and spatially resolved. For plasma-exposed surfaces, surface microstructure, chemical inhomogeneity, grain boundaries, etc., may determine optical, electric and chemical properties, hence the STM shows promise in elucidating the nature of such effects on the atomic or near-atomic scale. However, for rough surfaces, additional problems associated with data interpretation occur. For instance, if tip and surface have comparable roughness, then both tip and surface microstructures contribute to an STM image.

The aim of this talk will be to introduce workers in the field of plasma-surface interaction to the technique, to present some examples of microstructural investigations with the STM and their relationship to conventional methods, and to show a few examples of plasma-exposed and ion-bombarded surfaces studied using the STM.

[1] For a review of STM see, IBM J. Res. Dev. Vol. 30, Nos. 4/5 (1986).

APPLICATIONS OF PLASMA ETCHING

Hans W. Lehmann
Paul Scherrer Institute, c/o Laboratories RCA
CH-8048 Zürich

Plasma techniques have been used in industry for quite some time to treat surfaces and in particular to remove organic residues using O_2 -plasmas. It is now exactly 20 years ago since the first paper on applications of dry etching in the field of semiconductor technology was published(1). Although pattern transfer was not yet an issue at that time, it was already demonstrated that plasmas of fluorinated gases could be used for removing oxide films from silicon surfaces, for finding pinholes or forming scribe lines on Si-wafers. At about the same time work was started at TI, Bell Labs and IBM on using these plasmas for high fidelity pattern transfer in the semiconductor industry. It is particularly in this field where anisotropic plasma etching techniques have had their biggest impact; they have helped to revolutionize the field and make today's high density IC-chips possible. Plasma etching has replaced wet chemical etching in many different steps in IC-manufacturing. Furthermore, plasma etching is also of prime importance in the field of optoelectronics to transfer micron and sub-micron sized patterns to III-V materials. Further applications of dry etching pattern transfer techniques include among other optical diffraction (fabrication of very fine grating patterns), X-ray optics (Fresnel lenses) and micro-machining of magnetic materials for magnetic recording heads.

Most of these applications are based on high fidelity pattern transfer from a masking pattern formed by some lithographic technique into an underlying substrate. This transfer is preferably performed by an anisotropic etching technique. Anisotropic etch profiles are caused by the highly directional impact of ions onto the substrate surface, but there are a number of basic facts to be considered in order to optimize the pattern transfer process for a given material

or application. While directional ion impact is responsible for the anisotropic nature of the etched profile, it can also cause redeposition of physically sputtered material and be responsible for a deformation of the etch profiles due to faceting effects. Backscattering of sputtered species due to a small mean free path at the relatively high process pressure can cause a redistribution of etched material. On the chemical side of the etching process, the profile shape is influenced by the ease with which neutral species react with the material to be etched. While F atoms etch Si spontaneously (which can lead to undercut profiles), Cl only reacts with Si to any extent when the surface is subjected at the same time to ion bombardment. Often, special tricks have to be played to protect the sidewalls of etched profiles from attack by neutral species. Etching of narrow and deep trenches into single crystal Si for the next generation of VLSI-chips is a typical example where a lot of basic knowledge was required to optimize the process. Plasma etching in general can never be as selective as a purely chemical etching process because etching is always due to a subtle interplay between chemical and physical effects and the non-selective physical effects can never be completely excluded.

The parameter space in plasma etching is very large. Rate, anisotropy and selectivity often depend on subtle differences between etching systems. This not only makes process development extremely difficult and often limited to a given piece of equipment, but it also makes it close to impossible to study basic etching mechanisms on practical etching systems. Basic mechanisms, therefore, have to be studied in special beam systems where not only the influence of ions and neutrals but also energy and flux of particles can separately be investigated.

An important consideration for device and circuit fabrication in addition to etch anisotropy, is whether the etching process introduces damage in the material or causes degradation in the performance of the devices. Energetic

ions not only cause directional etching but devices subjected to ion bombardment can potentially also suffer from radiation damage. Furthermore, practically every plasma etching process also causes a certain degree of contamination either due to plasma polymerization or redeposition of sputtered material from chamber walls, etc. Ion damage and contamination can be minimized by using optimal etching conditions and good chamber design.

While more plasma etching systems used in manufacturing today are either single wafer parallel plate etchers or multi-wafer reactive ion etchers, there are currently a number of very promising techniques under closer investigation for possible use in production: Magnetron etching, microwave etchers in a variety of configurations, reactive ion beam etching or ion beam enhanced chemical etching. In addition, future etchers might also offer etching at a variable substrate temperature. So far, the only substrate temperature control which is used in industry is wafer cooling in order to prevent resist melting. But some recent publications indicate that temperature might be another real process parameter which so far has not been fully utilized.

1. Irving, S. M., Kodak Interface Proc. 2(1968) and US Patent No. 3 615 956 (1971) assigned to Motorola.

PLASMA-ENHANCED CVD OF SILICON-RELATED COMPOUNDS

W. A. Claassen and V. Rutten
Ned. Philips Bedrijven, Elcoma
Gerstweg 2, 6534 AE, Nijmegen, The Netherlands

The need for low temperature (<400°C) processes in the fabrication of integrated circuits had led to an increasing importance of thin films made by plasma-enhanced chemical vapor deposition. These layers are usually made in capacitively coupled reactors operating at frequencies between 50 kHz and 20 MHz. Basically, two reactor types are on the market, being the planar radial flow reactor, originally developed by Reinburg(1), and the hot-wall parallel-plate reactor(2). The compositional and structural properties of layers deposited in these types of reactors, depend, among other things, on the plasma parameters used. Due to electron impact on relatively stable molecules, active species, such as ions, excited molecules and radicals are formed. In these low pressure plasmas the mean electron energy is around 2eV, while the electron concentration is about 10^{10} cm^{-3} (3).

Confinement of the electrons near the electrode by using magnetic fields leads to an increase in the collision probability between electrons and reacting gas molecules. The influence of applying a magnetic field to a glow discharge on the layer properties has been studied recently(4). Besides electron cyclotron resonance (ECR) CVD methods a number of authors have studied remote plasma excitation and microwave-sustained plasma techniques to be used in the production of thin layers(5). It seems reasonable to expect that these new reactor concepts will lead to better film properties and/or higher throughputs.

Plasma silicon-nitride layers and, on a smaller scale, silicon-oxynitride layers are used for final passivation, because these layers act as barrier against diffusion of impurity ions such as sodium(6). Generally, these films are deposited from $\text{SiH}_4\text{-N}_2\text{-NH}_3$ gas mixtures at temperatures of about 300°C by

using RF-frequencies below 1 MHz. Plasma silicon-nitride layers deposited under these conditions contain about 15 to 25 at % hydrogen(7). Plasma silicon-nitride layers deposited by using low frequencies (<4MHz) show a compressive stress, which could lead to an unacceptable bow of the silicon substrate wafer. Furthermore, heat treatments after deposition could lead to cracks in the films, notably at steps.

The search for a layer which could be used for final passivation as well as for multilevel insulation has led to study plasma silicon-oxynitride layers(8,9). Incorporation of oxygen in the film, which can be achieved by adding N_2O to a $SiH_4-NH_3-N_2$ mixture leads to a strong decrease of the mechanical stress and to a reduction of the hydrogen content. Using SiH_4/N_2O mixtures plasma oxide layers are deposited, which are of interest for intermetal dielectrics, especially in high frequency devices.

As discussed above the various deposition parameters such as plasma conditions, temperature, pressure and gas phase composition largely influence the compositional, the mechanical and the electrical layer properties. We will discuss in detail the effect of the deposition parameters on layer properties of silicon-related compounds as deposited in capacitively coupled reactors. Besides the input gases given above silicon-nitride and silicon-oxide layers can be deposited by using SiH_4/NF_3 mixtures(10) or TEOS (tetra-ethyl-ortho-silicate)/ O_2 mixtures, respectively. Both alterations seem to have a positive effect on the film quality and will, therefore, be treated. Furthermore, topics such as film densification, layer adherence and silicon-(oxy) nitride/aluminium interactions will be discussed. The film properties of layers deposited in capacitively coupled reactors and of layers deposited in the microwave-, remote and ECR based reactors will be compared.

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4. See for example: M. F. Leahy and G. Kanowicz, Solid State Technol., 30, 99 (1987).
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HANDLING HAZARDOUS MATERIALS IN A PLASMA PROCESS

G. K. Herb
SEMATECH - AT&T Bell Laboratories

The good aspect about plasma processing is that patterns can be replicated from stencils with high fidelity and materials can be deposited with low temperature. However, the other side of the coin is that plasma operations usually have hazardous feed gases and chemical effluents as necessary parts of the process. The method chosen to deal with these corrosive, toxic, flammable, carcinogenic and/or high pressure gases and liquids may well determine the configuration of the plasma system, particularly the pumping package. Etching chemistries that rely on chlorine as the basic etchant species have particularly hazardous consequences. Deposition of materials from organo-metallic compounds should also have the utmost regard given to safe operation. The use of hazardous agent concentrating devices, such as cold traps and cryo-pumps should be avoided. Suggestions will be given on the method that may be used to define the plasma process configuration that pays the highest regard to safety for both the operator and the environment. Examples of real plasma processes will be illustrated that were discontinued, in spite of their utility, because of the unacceptable safety risk imposed by their continued operation.

LOW-ENERGY ION/SURFACE INTERACTIONS DURING FILM GROWTH FROM THE VAPOR PHASE:
EFFECTS ON NUCLEATION AND GROWTH KINETICS, DEFECT STRUCTURE, AND ELEMENTAL
INCORPORATION PROBABILITIES

J. E. Greene

Department of Materials Science, University of Illinois
1101 West Springfield, Urbana, Illinois, 61801 USA

Low-energy (≈ 20 -500 eV) ion bombardment of films during growth from the vapor phase plays an important and sometimes dominant role in controlling the growth kinetics and physical properties of films deposited by a variety of techniques such as glow discharge and ion beam sputtering, primary-ion deposition, molecular beam epitaxy utilizing accelerated beams, and plasma-enhanced chemical vapor deposition. Ion/surface interaction effects including trapping, enhanced adatom diffusion, preferential sputtering, collisional mixing, and surface segregation are used to interpret and model experimental results concerning the role of low-energy ion bombardment in altering nucleation and growth kinetics, elemental incorporation probabilities, and dopant depth distributions as well as in allowing the growth of new metastable phases.

During nucleation and the early stages of film growth on amorphous substrates, recent experiments carried out in UHV show that low-energy ion irradiation can lead to a more uniform distribution of islands, larger average island sizes, and an inhibition of secondary nucleation. On single-crystal substrates, experimental, as well as molecular dynamic simulations, indicate the feasibility of using ion/surface interactions to achieve "low-temperature" epitaxy.

Ion bombardment during deposition is often used to modify film microstructure. Depending upon the growth temperature, deposition rate, and collision dynamics, ion irradiation can either increase or decrease the defect concentration in as-deposited films. Experimental results from both polycrystalline and single-crystalline films will be used in conjunction with Monte Carlo and molecular dynamic simulations to discuss mechanisms leading to irra-

diation-induced film growth effects such as densification, the controlled alteration of dislocation densities, grain size, and preferred orientation, the interruption of columnar structure, and epitaxy.

Ion irradiation has also been shown to dramatically alter the chemistry of growing films and results will be discussed in terms of, depending upon the materials system and deposition conditions, preferential sputtering of alloy constituents, collisionally-induced dissociative chemisorption, and trapping processes. Examples of the latter case include the large increases in elemental incorporation probabilities, up to 8 orders of magnitude, and profile abruptness reported for accelerated-dopants in MBE Si.

THE PLASMA ENVIRONMENT IN THIN FILM DEPOSITION PROCESSES: A REVIEW

Donald M. Mattox

Surface and Interface Technology Division - 1834
Sandia National Laboratories, Albuquerque, NM 87185

In certain types of thin film deposition processes weakly ionized plasmas are used to enhance or enable the deposition process. These processes include: sputter deposition, ion plating, activated reactive evaporation, plasma polymerization and plasma enhanced chemical vapor deposition (PECVD). The plasma environment is used to provide the following:

- * Low energy ion bombardment and "activated" species to clean and desorb contaminants from a surface ("ion scrubbing").
- * Ions of inert species which may be accelerated to a surface to allow in-situ cleaning, heating, physical sputtering of surfaces and concurrent energetic particle bombardment during film growth to influence film growth and properties. Bombardment may also enhance reaction of adsorbed reactive species with the surface and depositing atoms.
- * Ions of reactive gaseous species which may be accelerated to a surface to enhance reactive deposition processes.
- * Ions of the film material which may be accelerated to the substrate surface to give them higher kinetic energy.
- * Incorporation of bombarding atoms into the depositing material to concentrations greater than the solubility limit.
- * "Activation" of reactive species to promote reactive deposition processes during film growth.
- * Fragmentation or partial disassociation of molecules of gaseous chemical compounds to provide condensible species.

A weakly ionized plasma ("processing plasma") is characterized by being a non-equilibrium plasma having a large fraction of un-ionized species. These un-

ionized species may influence the deposition process by providing a high flux of species to the substrate surface throughout the processing. The plasma may be sustained by the input of DC, RF or microwave energy and the electrons in the plasma may be influenced by a magnetic field in some cases ("magnetron processes"). Plasma-surface interactions generate a plasma sheath that accelerates low energy ions to surfaces giving a cleaning mechanism which may be termed "ion scrubbing".

In the plasma, various collision and attachment processes give rise to a spectrum of chemical species which may deposit on surfaces or be available to react with depositing species. These processes give rise to unique film materials such as amorphous Si:H or lowered reaction temperatures such as found in PECVD processing. The film deposition chamber may be the same chamber as the plasma chamber or it may be separate from the plasma chamber, relying on the long lifetime of "activated" or ionized gaseous species to allow them to be transported.

In the plasma environment high energy electron, ion and neutral bombardment may be intentional or unintentional, controlled or uncontrolled. The particles may be accelerated to the surface ("biased surface") or may be accelerated independent of the substrate surface by appropriate grid and electrode systems. In the case of ion bombardment of a surface at a low pressure reflected high energy neutrals may be an important source of energetic particles. In DC diode physical sputtering electrons and negative ions may be accelerated away from the cathode to give high energy particles that bombard the substrate. High energy electron bombardment may be used to heat a surface.

High energy ion bombardment may be used to heat the surface, remove surface material by the momentum transfer process of physical sputtering or by chemical reaction and vaporization from the surface ("chemical sputtering"). Energetic particle bombardment during film deposition may promote reactive deposition processes when using reactive species or modify the properties of the deposited

film material. Among the film properties that can be modified by concurrent bombardment are: residual stress, film morphology, density, hardness, wear resistance, chemical etch rate, optical properties, impurity/solute incorporation, surface coverage and environmental stability. Some of these properties are very sensitive to the energy and flux of concurrent energetic particle bombardment. Many of these properties are interdependent.

Species to be deposited in the plasma environment may originate from the sputtering process (sputter deposition), from thermal vaporization, arc vaporization or from gaseous chemical species introduced into the plasma (chemical ion plating, PECVD).

The species and energy of the bombarding species, nature of the adsorbed species and the ratio of the flux of bombarding energetic particle to the flux of depositing particles are important process variables in film deposition processes performed in a plasma environment.

PLASMA-ASSISTED DEPOSITION OF POLYMERS

Riccardo d'Agostino
University of Bari - Department of Chemistry
via Amendola 173, 70126, Bari, Italy

The main goal of this overview of plasma polymerization is to show how it is possible to control the physical and chemical characteristics of polymer thin films by means of the chemistry of the process and reactor parameters.

The main classes of monomers generating the various types of films will be listed. More emphasis will be given to fluorine containing polymers, organosilicons, metal-polymer composites and hydrocarbons. All these classes of films will be considered as individual case studies by trying to define the mechanism of polymerization and the parameters affecting the chemical structure, the cross-linking density and the properties of the films. In particular, it will be shown the effect of the discharge frequency, reactor geometry, superimposed magnetic field, substrate bias, substrate temperature, power input, and pressure. An effort to draw general trends will be made.

The review will conclude with a section on plasma polymer applications. This part will be subdivided into a section on applications in which the surface is of primary importance and one in which the bulk properties of the film is the determining factor.

PLASMA MEASUREMENTS IN A MAGNETRON SPUTTERING DISCHARGE

J. B. Almeida, F. Guimarães, M. M. D. Ramos
Universidade de Minho
Laboratório de Física
P-4719 BRAGA Codex

The authors have for some time been involved in the design, fabrication and use of DC magnetron sputter coating systems for research purposes, some of which have previously been reported.

This presentation is concerned with the physical characterization of the discharge plasma in one of those magnetrons, in order to improve the design of future versions.

The magnetron is of the planar type, with circular symmetry, and uses a DC voltage of a few hundred volts to sustain a discharge in an atmosphere of argon at a pressure of around 4×10^{-2} torr, assisted by a magnetic field created by a permanent magnet.

In order to characterize the processes in the magnetron, the distribution of the magnetic field has been measured and so have several parameters of the discharge.

By means of single and double electric probes, the authors have measured the potential distribution and the density of charged particles and have drawn conclusions in respect of the speed distribution of electrons in the plasma.

The results of the experiment are show in the body of the presentation and are used to recommend improvements for the future magnetron designs.

KINETICS OF A LOW-PRESSURE H_2 MULTIPOLE DISCHARGE USED FOR GaAs TREATMENT

J. Bretagne*, D. Jacquin*.** and R. Ferdinand*.**

*Laboratoire de Physique des Gaz et des Plasmas

Unité Associée du CNRS, bâtiment 212

Université Paris-Sud 91405 ORSAY CEDEX, France

**Laboratoires d'Electronique et de Physique Appliquée,
3 Avenue Descartes, BP15
LIMEIL-BREVANNES, France

This work is devoted to the study of kinetic equilibrium between electrons, H_2 molecules, H atoms and positive H^+ , H^+ and H^+ ions in a multipole discharge working in low-pressure (1-15mTorr) and moderated current conditions. In this situation, the electron energy distribution function (e.e.d.f.) is largely non-maxwellian. The kinetic model that determines the densities of the plasma species is coupled to the e.e.d.f. which is self-consistently calculated through the use of the Boltzmann equation. The dependence of these densities on the experimental parameters is studied. The influence of the main processes, volume ones and those involving plasma walls is emphasized. A comparison of the results of the modeling with experimental ones is attempted through the analysis of the spectra of Balmer lines. From the Doppler profile of these lines, we identify the processes leading to the formation of H^* excited states and determine the H and H^+ densities. Probe techniques are implemented to control the plasma parameters.

ITERATIVE DATA ANALYSIS OF LASER-INDUCED FLUORESCENCE SIGNALS

John Goree

Department of Physics and Astronomy, University of Iowa
Iowa City, IA 52242

Laser-induced fluorescence (LIF) is a widely used diagnostic for observing ions and neutrals in plasmas, including those where ions play a dominant role in plasma surface interactions. An important application is the measurement of ion velocity distribution functions. A tunable dye laser is pointed at a plasma and its frequency is scanned while observing fluorescence. Ideally, the fluorescence signal plotted versus frequency would give a direct measurement of the distribution function as a result of doppler broadening; however, several additional mechanisms broaden the spectral line. The resonance width of the excitation transition in the LIF scheme will always be a factor. When a laser with more than 100 MHz bandwidth is employed, instrumental broadening must also be considered, and when a powerful pulsed laser is used, saturation broadening must be taken into account. A method of data analysis for determining the ion distribution function is described. The LIF signal for a set of plasma and laser parameters is computed, including all the broadening effects, and plotted against frequency. The experimenter selects a set of plasma parameters, compares the resulting computer calculation to his data, and then iteratively adjusts the plasma parameters until the calculated curve fits the data.

OXYGEN PLASMA ETCHING OF MULTI-LEVEL RESISTS

M. A. Hartney

Dept. of Chemical Engineering, UC Berkeley

The relative roles of ion bombardment, oxygen atoms and neutral oxygen molecules have been examined for plasma etching of common photoresists and silicon containing resists. A reactor built in our laboratory is equipped with a quadrupole mass spectrometer and a cylindrical mirror analyzer which allows measurement of the ion energy distribution during etching. In addition, the mass spectrometer is used for species identification and to determine the extent of oxygen dissociation in the plasma.

The degree of dissociation was measured as a function of reactor pressure (between 20 and 80 mtorr) and input power (between 0.12 and 1.85 W/cm²). Dissociation decreases with increasing pressure, while the etch rate of standard resists increases. In addition, ion energy and current decrease with increasing pressure, therefore the undissociated molecules are the limiting species in etching under these conditions.

Experiments were performed to determine the contribution of ion induced damage to the etching of resists. The effect of damage is minimal for etching hydrocarbon resists, but it enhances the formation of an oxide layer when etching silicon containing polymers.

ELECTRON BEAM AND LASER BEAM MACHINING PROCESS

M. Akkurt

Process, characteristics, equipment, and procedures for electron and laser beam micromachining of solids will be discussed.

ION BEAM ETCHING AND PLASMA ETCHING IN STRUCTURING ELECTRONIC DEVICES

K. Fischer

AEG S 13, Theresienstr., 2, D-7100 Heilbronn

W. Möhl

Technics Plasma GmbH, D-8011 Kirchheim bei München

In connection with the short talk about "A novel microwave ion beam-system for surface processing" this paper describes principles and practical applications of ion beam milling and plasma etching.

The explanation are based on the exchange of experiences between the company Technics Plasma GmbH and companies, which are using the plasma and the ion beam etching. One example is the ion beam milling of cadmium-mercury-telluride, which is patented in the USA and Germany. Cadmium-Mercury-Telluride is a material, which is not allowed to be heated more than 120°C.

There will be given a summary of requirements for the production of semiconductors for the conditions of rooms and climate, the changes in chemicals, the masks, the discs, the exposure and the development. The necessity of new and revised methods is easily realized by the complicated structures of the microprocessors.

The requirements and the data for the ion beam and the plasma-etching are given in tabulated summary. The quality of ion beam and plasma etching are shown by examples.

There will be listed the different applications as for example:

- Pre-treatment of glass, ceramic or semiconductor substrates to improve the adhesion of metal coatings or to improve bonding qualities.
- Quick preparation of synthetic parts to improve surface quality for printing or glueing applications.
- Photoresist stripping, even after implantation, sputtering, R.I.E., R.I.B.E. etc.

- Cleaning of substrates.
- Cleaning of hybrids circuits.
- Desmearing of PC boards.
- Etching of polyimide.

In the talk this list will be completed.

One important point will be the discussions about the collaboration between the groups of different countries.

The oral presentation will be given by K. Fischer.

LOW-ENERGY ACCELERATED-ION DOPING OF Si DURING MOLECULAR BEAM EPITAXY:
INCORPORATION PROBABILITIES, DEPTH DISTRIBUTIONS, AND ELECTRICAL PROPERTIES

L. C. Markert, J.-P. Noel, and J. E. Greene
University of Illinois, USA

J. Knall, M.-A. Hasan, and J.-E. Sundgren
Linköping University, Sweden

Ion-surface interactions can change film growth kinetics dramatically. During molecular beam epitaxy (MBE) of Si, many common dopants present problems due to low incorporation probabilities and/or strong surface segregation. Our experiments using low-energy accelerated-dopants during Si MBE growth demonstrate increases in elemental incorporation probabilities by several orders of magnitude, improved control over concentration depth distributions, and substitutional incorporation at concentrations exceeding equilibrium solid-solubility limits. These results stem from effects such as trapping and creation of preferential adsorption sites due to low-energy ion bombardment.

TREATMENT OF POLYMERIC FILMS (PP, PET) BY A NON-EQUILIBRIUM LOW PRESSURE
PLASMA OF NH₃, N₂ AND Ar

F. Arefi, V. Andre, F. Tchoubineh, P. Montazer-Rahmati,
J. Amouroux, M. Goldman*
Laboratoire de Génie Chimique: Equipe des Réacteurs en Phase Plasma
ENSCP, 11 rue P. & M. Curie, 75231 PARIS Cedex 05

The surface treatment of polymeric films (PP, PET) has been realized in an installation in which a background pressure of 10^{-4} Pa can be obtained in order to eliminate partly the adsorbed or condensed gases on the films. The plasma treatment is carried out in NH₃, N₂ or Ar, and is meant to improve the adhesive properties of the film towards metallic coatings.

The treatment process includes a hollow (high-voltage) electrode, parallel to the grounded cylinder on which the polymer film is enrolled. The rotational velocity of the cylinder (0.8 to 22 m/min) defines the treatment time of the film under the glow discharge. The industrial generator employed (70 kHz, 800 W max. power) gives rise to a glow discharge for the range of pressure varying between 0.01 to 0.08 Pa.

The control of the treated samples has been possible through methods of analysis including ESCA, contact angle measurements (surface energy calculation) as well as the structural analysis by TEM.

The analysis of the surface free energy is accomplished by the use of an image processing system which allows an automatic measure of the contact angle every 0.6 seconds. This process presents the advantage of obtaining the dispersive and polar components of the surface tension with a speed compatible with the kinetic measure of the surface modification of the treated films. The measurements show an increase of the dispersive component of the surface tension for treatment times below 1 second, that is to say for the treatment for which no nitrogen moieties have been detected by ESCA analysis (in the case of plasma

treatment in NH_3 and N_2). This leads us to propose the hypothesis that we might have a rotation of the polymeric chains rather than a grafting of the polar nitrogen groups on to the surface for short treatment times. The increase of the polar component can be pointed out only for longer treatment times (>0.46 sec.), this phenomenon is accompanied with a decrease of the dispersive component.

The understanding of the aging process of the samples has been possible with the help of the contact angle method. This method is more appropriate for the analysis of the surface properties, than ESCA, particularly to appreciate the evolution of the dipoles and the surface free energy.

The future analysis will be related to the study of the structure of the metals which are deposited onto the pretreated surface as well as the nature of the metal-polymer interface; this will be possible with methods such as Auger, ESCA and TEM.

*Laboratoire de Physique Des Décharges. ESE-CNRS, Plateau du Mouton 91190 Gif Sur Yvette.

PLASMA ETCHING OF Si-Ge AMORPHOUS THIN FILMS DURING GROWTH BY
REACTIVE SPUTTERING

M. Androulidaki, P. Tzanetakis, Y. Fragiadakis
F.O.R.T.H.-Research Center of Crete
Institute of Electronic Structure and Laser
P. O. Box 1527, GR 711 10 Iraklion, Crete, GREECE

A series of hydrogenated amorphous Si-Ge alloys with varying Ge composition and hydrogen content have been grown by reactive sputtering under various plasma conditions at substrate temperatures between 150°C and 250°C.

Some films were post-deposition plasma treated at the same temperature as during growth in H₂ and/or Ar.

The hydrogen content and bonding in the films was determined by IR spectrometry and their optical and photoelectronic properties were measured.

The growth rate was found to increase drastically with Ge content. An attempt is made to clarify the role of plasma etching during growth in this system.

ION BEAM ASSISTED ETCHING: DAMAGE MECHANISM

A. Bensaoula and A. Ignatiev
University of Houston, USA

We have studied the etching behavior of tungsten (W), molybdenum (Mo) and tungsten silicides (WSi_6) under various experimental parameters. Both the spontaneous and the ion beam assisted etching of these thin films with XeF_2 were investigated with AES and XPS. The principal mechanism responsible for the etch is found to be defects present near the surface of the films and the enhancement under inert gas ions is mainly due to damage generated by these energetic particles. A model of the etch will also be presented.

EFFECT OF PARTIAL ORIENTATION OF Cu^{2+} - COMPLEXES IN $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$

A. Bonanno, M. Camarca, R. Bartucci, L. Sportelli, E. Colavita
Dipartimento di Fisica, Università degli Studi della Calabria
87036 Arcavacata di Rende, Cosenza, Italy

G. Balestrino, S. Barbanera
Istituto di Elettronica dello Stato Solido
Consiglio Nazionale delle Ricerche
Via Cineto Romano 42, 00156 Roma, Italy

Electron Paramagnetic Resonance line shapes of Cu^{2+} ion centers in the new high- T_c $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ superconductor are calculated and compared to the experimental data by a program of best fitting. The partial orientation of paramagnetic complexes previously observed, is computer simulated. As a result of the simulation the less abundant cupric component gives the largest signal in the g region.

Introduction

The new high- T_c superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ has a tripled perovskite structure with two non-equivalent Cu^{2+} ion sites in the unit cell [1]. Actually, the less abundant cupric complex, $\text{Cu}(1)$, has a rectangular planar environment characterized by rows of oxygen atoms along [010] directions and by rows of oxygen atom vacancies along [100] directions. The most abundant, $\text{Cu}(2)$ ion site has, instead, a pyramidal oxygen-coordination. Since theoretical considerations suggest that copper oxygen planes are responsible for the superconductivity while copper-oxygen chains may not be essential, we tried to distinguish between the two contributions versus temperature in Electron Paramagnetic Resonance (EPR) spectra.

Discussions and conclusions

The EPR signal[2] of Cu^{2+} ion sites in Y-Ba-Cu-O was computer simulated taking into account only the Zeeman interaction of the electron spin with the external magnetic field and the hyperfine coupling of the unpaired electron with the nuclear spin. Of course, the variation of the transition probability with

the g-value is considered, while the magnetic hyperfine orientations of the z-axis of the paramagnetic microcrystals with respect to the magnetic field, but a partial orientation of paramagnetic complexes was necessary to reproduce the experimental data. On the other hand, also angle-resolved EPR measurements [3] on pellets show an angular dispersion of some features. Fig. 1 shows the comparison between the experimental (full line) and calculated (dashed line) EPR spectrum at $T = 90\text{K}$. In the insert it is reported the calculated powder EPR spectrum (dashed line), that is the envelope of line shapes from random oriented paramagnetic microcrystal axes with respect to the magnetic field. The presence of oriented domains in the pellet appears to be important because it is almost 20% of the whole material. Since a large number of bondings lie in the plane perpendicular to the z-axis of the unit cell, we expected a strong signal in the g region as actually occurs, but the present analysis shows that it comes essentially from the less abundant Cu(1) cupric component. Both absorptions, instead, contribute in the g-region. In conclusion, the computer analysis revealed the existence of oriented domains in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ pellets and the contribution of the most abundant Cu(2) ion sites which give broader and less intense responses to the magnetic probe than Cu(1) cupric complexes.

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THEORETICAL ANALYSIS OF THE INFLUENCE OF FOIL INHOMOGENEITIES ON THE ANGULAR VARIATION OF THE ENERGY-LOSS

N. E. Capuj* and M. M. Jakas**
Centro Atomico Bariloche, C.N.E.A.
8400 Bariloche, Argentina

Analytical calculations have been done in order to evaluate the effects of foil inhomogeneities on the angular variation of the mean energy-loss for light swift ions after traversing thin solid films.

By introducing a scaling similar to that used in multiple scattering, and assuming that inhomogeneities are small compared to the mean thickness we can obtain an expression which results independent of the ion, ion-energy, and target specie and inhomogeneity.

Comparisons with previous Monte Carlo calculations show excellent agreement. Analysis of experimental results, in cases where information of the target inhomogeneity area available, indicate that, although not all angular variations are caused by inhomogeneities, there are cases where they can, to a great extent, be contributing to the mentioned effect. It must be thus concluded that, in experiments of this kind, an estimation of the target inhomogeneities should be necessarily included.

*Holder of a fellowship of CONICET, Argentina

**present address INVAP S.E., Moreno 1089, 8400 Bariloche, Argentina

THIN FILM INHOMOGENEITY CHARACTERIZATION BY ION BEAM TECHNIQUE

N. E. Capuj*, N. R. Arista, G. H. Lantchsner, J. C. Eckardt, and M. M. Jakas**

Centro Atomico Bariloche, C.N.E.A.

8400 Bariloche, Argentina

A method to evaluate thin film inhomogeneities (density and thickness fluctuations) is presented.

This method is based on the fact that the energy straggling for rough foils has two contributions, one of intrinsic origin¹, which varies linearly with average thickness $\langle t \rangle$, and a second contribution due to the thickness fluctuations², which varies with $\langle \Delta E \rangle^2 \propto \langle t \rangle^2$. If we change the average thickness $\langle t \rangle$ by tilting the foil an angle θ_f each contribution changes in a different way with θ_f .

Using the measurements of mean energy-loss $\langle \Delta E \rangle$ and FWHM of the energy spectra of ions traversing a thin foil tilted at two different angles θ_1 and θ_2 , we can evaluate the roughness coefficient defined as $\rho = \sigma / \langle t \rangle$, where σ is the standard deviation of the thickness distribution.

Here, we present the first results for 180A Aluminum foils with $\rho = 0.09 \pm 0.12$.

¹N. Bohr, Mat.-fys. Medd., Acad. Copenhagen, 28, No. 8, (1948).

²F. Besenbacher, J. U. Andersen and E. Bonderup, Nucl. Instr. and Meth. 168, 1-15 (1980).

*Holder of a fellowship of CONICET, Argentina.

**Present address INVAPO S.E., Moreno 1089, 8400 Bariloche, Argentina.

PLASMA INDUCED POLYMERIZATION STUDY OF THE INTERFACE PLASMA-POLYMER

F. Epailart and J. C. Brosse

Laboratoire de Chimie et Physicochimie Macromoléculaire,
Unité Associée au CNRS n° 509 - Faculté des Sciences -
Route de Laval - BP 535 - 72017 LE MANS CEDEX France

J. Bretagne and A. Ricard

Laboratoire de Physique des Gaz et des Plasmas,
Université de Paris-sud, 91405 ORSAY France

Multifunctional acrylates can be polymerized under a cold plasma treatment when the monomer is spread on a substrate and subjected, for example, to a tetrafluoromethane plasma. The generator used in this study is a micro-waves generator (433 Mhz). The polymerization rate and the resulting polymer structure are related to the conditions of plasma generation. The chemical structure of the polymer surface and subsurface has been characterized by ESCA and FTIR-ATR. The plasma emission in the uv-visible region has been recorded and the excited species F^* , CF^* , CF_2^* emission are observed during the plasma induced polymerization at the surface of the growing polymer or in the gas phase. A mechanism is proposed from comparison between the excited species concentrations in the gas phase, at the polymer surface and the concentration of CF_2 groups bound to the polymer skeleton at its surface and in the bulk.

ION IRRADIATION INDUCED DESORPTION FROM STAINLESS STEEL
IN FUSION REACTORS AND OTHER PLASMA DEVICES

I. H. A. Filius
Interfaculty Reactor Institute, The Netherlands

In nuclear fusion reactors and other plasma devices ions escaping from the plasma may release molecules that are adsorbed at the surfaces of construction materials.

The released molecules can interact with the plasma and may thus have a considerable influence on the plasma characteristics.

Results will be presented on the desorption process of H_2 , O_2 , OC , CO_2 and CH_4 adsorbed on 316L stainless steel, irradiated with light ions.

THE TRANSFER OF DRY ETCHING PROCESSES BETWEEN DIFFERENT EQUIPMENTS

A. J. Hydes, T. I. Cox, D. A. O. Hope and V. G. I. Deshmukh

Royal Signals and Radar Establishment
St. Andrew's Road, Great Malvern,
Worcestershire WR14 3PX, United Kingdom

One of the most serious problems facing the process engineer is the irreproducibility of etch performance in dry etching. It has become evident that, in order to alleviate this problem and so facilitate the process transfer between different machines, it is necessary to fully characterize the plasma system. We have conceptually divided this into two tasks (1) to characterize the glow discharge, and (2) the glow-sample interface characterized by the D.C. bias. For these R.F. plasmas, operating at 13.56MHz, the D.C. bias is measured between the sample and the plasma by a voltage probe, to which is added the plasma potential obtained from Langmuir probe measurements. The glow can be characterized from a knowledge of the partial pressures of ground state species and the electron energy distribution function, as measured by a Langmuir probe (LP). This is, however, an invasive technique and is not simple to operate. Optical emission spectroscopy (OES) on the other hand, is non-invasive, simple to run and has been shown, in our studies of Argon plasmas¹, to yield corroborative and complementary information on the electron energetics of the plasma. We have used our diagnostic techniques of OES, to assay the glow discharge, and a LP and a voltage probe to characterize the DC bias, in order to try to set up the same plasma in two very different etching machines. The purpose was to determine whether a dry etch process could be transferred between etching machines by the physical characterization of the important plasma parameters. The process studied was the etching of polyimide by an oxygen RIE plasma and the one response of the process to be transferred was the anisotropy of features etched in the polyimide through a thin (1000Å) non-erodable aluminum mask.

Our results demonstrate that a dry etch process can be successfully transferred between two different etching machines using the plasma characterization method, which should be applicable to other more complex systems. It is important to note, however, that the LP measurements showed that the plasmas in the two machines were markedly different in respect to electron densities, and that these scaled approximately with the magnitude of the etch rates in each machine. This fact, together with the observation that our optical fingerprinting technique is insensitive to plasma density, suggests that, although we set up similar plasmas in the two etching machines, they differed in the absolute magnitude of the concentration of species.

- 1) Cox, T. I., et al., J. Phys. D20,820,1987.

OPTICAL EMISSION SPECTROSCOPY RESULTS CORRELATED WITH CURRENT DISTRIBUTIONS IN THE VICINITY OF THE SUBSTRATE DURING MAGNETRON SPUTTERING OF Ti

T. Pech and A. Ricard
L.P.G.P. Universite Paris-Sud
Batiment 212, 91405, Orsay Cedex, France

In Ar discharges at 0.27 Pa, the spatial variations of the $I_{375.3}$, $I_{750.4}$ and $I_{375.9}$ emission intensities related to the excited states of neutral Ti and Ar, as well as of ionized Ti, respectively, have been investigated in the 0 to 15 mm distance range from the substrate. In order to characterize the discharge conditions prevailing at various selected magnetron current values (I_m up to 4 A), the currents flowing into the substrate, as well as into the grounded guard ring around it, and into the grounded rotatable shutter have been simultaneously measured during the runs. The experiments have been carried out with the substrate being either grounded or negatively biased (up to $\sim 750^\circ\text{C}$), and at variable temperatures (up to $\sim 500^\circ\text{C}$).

Analysis of the currents measured at the various locations reveals predominantly electron or ion currents flowing into the various electrodes, according to the prevailing experimental situation. The correlations found between the variations in nature and in magnitude of these currents on one hand, and the variations in space and in magnitude of the emission intensities, on the other, suggest that the electron density n_e distribution in the inhomogeneous plasma in the vicinity of the substrate and farther away from it undergoes considerable modifications according to the variable experimental parameters. It turns out that the reduced $I_{375.3}/I_{750.4}$ emission intensity values may be far more accurately related to the density of the sputtered Ti, than the crude $I_{375.3}$ values. The so reduced intensity values obtained at 15 mm from the substrate for the various I_m values are practically independent of the other largely variable experimental parameters. It seems that consideration of similar reduced emission intensity values may find a more general justification for sputtering systems where the presence of grounded parts in the vicinity of the substrate leads to substantially modified n_e distributions for variable experimental situations.

CERAMIC PLASMA SPRAYED COATINGS AND ITS APPLICATIONS

E. Munduate

Plasma spraying technology can be considered as a development of traditional thermal spraying methods. This technique has made it possible to deposit a large variety of materials onto different substrates in an economical way.

In this paper the process and equipment are briefly described. Most of the work reviews some interesting applications of this technique. These include thermal barrier coatings made of ceramic materials (TBC) for engine components. The materials and structures of such coatings are analyzed. The properties of the coatings in relation to the problems found in service conditions are described. Finally, some laboratory experiences showing the thermal shock resistance of the coating are described.

REACTIVE ION BEAM ETCHING STUDIES OF TUNGSTEN WITH CF₄ USING ION SCATTERING SPECTROSCOPY

T. I. Cox
Royal Signals and Radar Establishment
St. Andrew's Road, Great Malvern
Worcestershire WR14 3PS, United Kingdom

Reactive Ion Beam Etching (RIBE) is a real etching technique which allows the study of some ion surface interactions which are of importance in semiconductor processing.

In this work the ion beam is generated in a Kaufmann source and the ion energy may be varied between 100 and 1800 eV. The positive ions emitted from the bombarded samples are energy and mass analyzed using an electrostatic energy analyzer followed by a quadrupole mass filter. This system allows observation of positive ions formed in three ways:

- i) By ionization of background gas molecules caused by collision of background molecules with energetic ions in the primary beam. The observed energy of these ions gives a measure of the potential at the point of formation in the ion beam.
- ii) By formation of secondary ions which are emitted from the sample surface. This yields information on the products of the etching reaction and also on the chemical composition of the bombarded sample.
- iii) Primary ions may be scattered from the surface. If noble gases are added to the source then the energy spectrum of these scattered ions (e.g., He⁺) yields information on the elemental composition of the surface via Ion Scattering Spectroscopy.

These techniques have been applied to a study of the RIBE of tungsten in an ion beam generated from CF₄. The ions generated by the three mechanisms are found to have different energy distributions which therefore allows the separate

observation of these three groups of ions. It is found that for low beam energies ($<1000\text{eV}$), a large fraction of the tungsten surface is covered with fluorine. The principal products in the SIMS spectrum are W^+ , WF^+ , WF_2^+ and C^+ . These results will be discussed in terms of a model for the fluorocarbon RIBE of tungsten.

HIGH POWER FACILITY FOR INVESTIGATIONS OF LASER SUPPORTED PLASMA-SURFACE INTERACTION

G. Sliwinski

Polish Academy of Sciences, Institute of Fluid-Flow Machines, Poland

A high-power facility for investigations of laser supported plasma-surface interaction is presented with particular attention to the experimental potential of two independently driven processing stands, i.e., vacuum chamber and optical plasmathron. Results of plasma-surface interreaction experiments are discussed.

SURFACE ANALYSES OF CORONA TREATED POLY(ETHYLENETEREPHTHALATE)

Y. De Puydt, B. Bertrand

Catholic University of Louvain-la-Neuve, PCPM Laboratory
1, Place Croix du Sud-B 1348 Louvain-la-Neuve, Belgium

Y. Novis, R. Caudano

Facultés Universitaires Notre Dame de la Paix, LISE Laboratory
61, Rue De Bruxelles - B 5000 Namur, Belgium

P. Lutgen

DuPont de Nemours s.a.
L 2984 Luxembourg, G. Duchy of Luxemburg

Poly(Ethylene Terephthalate) (PET) Mylar samples have been treated by corona discharge in order to improve their adhesive properties. The corona treatments have been performed in different atmospheres including nitrogen, ammonia and air.

XPS has been used to investigate the chemical modifications occurring at the PET surface after these corona treatments. XPS results show that nitrogen incorporation takes place as non-oxygenated nitrogen functionalities, like amine or cyano groups. These are present at the surface of all the corona treated samples, but in different concentrations depending on the gases used in the corona discharge. Furthermore, XPS analyses performed after heating of the treated samples show a higher thermal stability of the corona induced surface modifications in the case of nitrogen and ammonia.

ISS and statis SIMS analyses have also been performed due to their higher surface sensitivity as compared with XPS: ISS reveals that nitrogen is not present at the top most surface layer of the treated samples but is incorporated just beneath. At the top surface, the samples present an oxygen rich composition. Finally, static SIMS spectra show that corona treatment induces more pronounced surface degradation when performed in air than when in nitrogen or ammonia.

These results are discussed in relation with adhesive properties of PET.

RBS AND RNA STUDY OF PLASMA OXIDATION OF SILICIDES

A. Climent-Font, J. Perrierè and A. Straboui

We have shown the possibility of growing thick oxide films on refractory metal silicides by plasma oxidation in the 500 to 900°C temperature range. Thin layers of Si rich silicides TiSi_x and WSi_y ($x, y > 2$) deposited onto Si or SiO_2 by cosputtering, have been oxidized in an rf plasma at floating potential.

The composition and oxide thickness, and the oxide growth rate were determined by the complementary use of nuclear reaction analysis and RBS which was also employed to obtain the depth distribution of cations in the oxide, and the changes in the stoichiometry of the silicides.

We have found that the plasma oxide growth on silicides can be schematically described by a two-step process. (i) First, it appears that the oxygen atoms incorporated during the plasma treatment form a pure SiO_2 overlayer. These SiO_2 metal free films on the WSi_y films are formed by Si atoms in excess in the silicide until the composition WSi_2 is reached. The nature of the substrate, that is Si or SiO_2 , does not seem to have any effect. For the TiSi_x films on SiO_2 the behavior is similar to the WSi_y films, but the final composition of the silicide lies between TiSi and TiSi_2 . Moreover, when the substrate is pure Si, incorporation of Si atoms from the substrate takes place and the amount of Si in the silicide is always higher than the one giving TiSi_2 . (ii) Further oxidation yields a high rate of oxygen atoms incorporation. At the same time, the depth distributions of cations obtained by RBS analysis show the presence of Ti or W cations in the oxide. Thus, in this regime the silicide itself is oxidized leading to the formation of an oxide mixture.

Under the same conditions of oxygen pressure, temperature and duration of the treatment, but without the plasma, the thermal oxidation does not yield to significant oxide growth. As the plasma oxidation was carried out at the

floating potential, no net current passes through the sample during the oxidation. This situation is quite different from the plasma anodic oxidation, and thus, this means that this oxide growth can be related to the presence in the plasma of appreciable concentrations of atomic oxygen. This species is known to be very reactive and can induce some surface phenomena allowing for its injection into the oxide. This could be the origin of the observed enhancement with respect to the thermal growth.

These findings will be compared to the plasma oxide growth on Si and the kinetics of the oxidation process will be analyzed in the frame of the classical laws of oxide growth.

GLASS SUBSTRATE CHEMICAL ANALYSIS DURING SPUTTER DEPOSITION
BY GLOW DISCHARGE INDUCED X-RAY SPECTROMETRY

H. Hecq
Laboratoire de Chimie Inorganique et Analytique
Universite de Mons
23 Ave. Maistriau 7000 Mons Belgique

A new technique is presented that allows the study in situ of the chemical composition of films and substrates during deposition. A sputtering chamber is coupled with a vacuum x-ray spectrometer allowing the analysis of x-ray emission induced by the fast electrons of the sputtering discharge (rf or dc mode). The chemical composition of glass substrate during C deposition is determined. In the case of alkali glass substrate, a variation of the surface concentration with time deposition is shown. Quantitative chemical analysis is demonstrated.

EFFECT OF MODULATION ON THE PLASMA DEPOSITION
OF HYDROGENATED AND FLUORINATED SILICON NITRIDE

Grazia Cicala
Centro Di Studio Per La Chimica Dei Plasma
Universita di Bari
70216 Bari, Italy

and

Daniel L. Flamm, Dale E. Ibbotson, John A. Mucha
AT&T Bell Laboratories
600 Mountain Avenue, Murray Hill, NJ 07974, USA

Silicon nitride is used as a passivation coating, an interlevel insulator, a dielectric coating for optical applications and, occasionally, as a gate dielectric. However, conventional plasma-deposited silicon nitride, made from various combinations of $\text{SiH}_4/\text{NH}_3/\text{N}_2$ and inert gas carriers, contains up to 30 atomic % hydrogen; this hydrogen causes instability in MOS devices by creating traps near the gate oxide. Researchers generally believe that the trouble originates from Si-H bonds. Fluorinated silicon nitride films, deposited in both 14 MHz and 200kHz discharges, have been shown to minimize these effects and exhibit superior electrical and optical properties that compare favorably to thermal CVD silicon nitride.

In this study hydrogenated and fluorinated silicon nitride films were deposited from $\text{SiH}_4\text{-NF}_3\text{-He}$ mixtures in a novel resonant reactor operated at 18MHz and 350°C. The discharge was (square-wave) modulated with a 50% duty cycle between 0 and 10kHz, in order to vary the supply of reactive species relative to the residence time in the reactor. Film compositions were characterized by infrared spectrometry and Rutherford backscattering, while refractive index and thickness were measured with a prism coupler. Time resolved emission actinometry was used to examine the excitation of selected species as a function of phase

some CW and modulated discharges. This modulation influences the deposition rate and film properties, and can greatly improve the uniformity of deposition along the flow direction. We will discuss possible mechanisms for these effects.

ESTIMATION OF STRUCTURAL DAMAGE INDUCED BY TECHNOLOGICAL PROCESSES ON SURFACE
OF CRYSTALLINE BINARY COMPOUND BY X-RAY PHOTOELECTRON DIFFRACTION: APPLICATION
TO REACTIVE ION ETCHING OF GaAs (001) SURFACES

J. Olivier and P. Alnot
Thomson-CSF, LCR, Domaine de Corbeville, 91401 Orsay, France

Due to photoelectron diffraction in the atomic network, the measurement of core electron intensity or Auger electron intensity from a single crystal surface as a function of electron take-off angle gives rise to plots where pronounced fine structure is superimposed on the instrumental response function[1]. This anisotropy of the XPS signal is maximum for a perfectly crystalline surface and zero for amorphous material. Great care should be taken in quantitative interpretation concerning the surface stoichiometry and chemical composition after different treatments of III-V compounds[2].

The degree of structural damage induced by chemical or ionic processes on crystalline binary compound is estimated by means of X-ray photoelectron diffraction using a model where the damaged layer, out of stoichiometry, is assumed to be homogeneous and in the amorphous state[3]. The experimental angular distribution curves (ADC's) fitted from reference crystalline ADC's give an estimation of the disordered surface layer thickness and composition.

We investigate the surface composition and structural damage suffered by GaAs (001) consequently to reactive ion etching (CF₄, 440kHz) of GaAs-Si₃N₄ interfaces. This process is used to open a channel through the passivating layer before gate metal deposition. The most striking results show that the reaction proceed through the formation of a GaFx reaction layer, the thickness of this layer increasing with exposure. At the interface between GaFx and GaAs we observed the formation of an As amorphous layer. Also we have seen that the plasma induces structural damage within GaAs lattice which can be partially suppressed through annealing (5mn at 400°C).

- 1) C. S. Fadley, Progress in Surface Science, 16(1984)275.
- 2) P. Alnot, J. Olivier, F. Wyczisk and C. S. Fadley, J. Electron Spectrosc. Relat. Phenom., 43(1987)263.
- 3) J. Olivier and P. Alnot, submitted to Semicond. Sci. Technol.

IN-SITU XPS STUDIES OF THIN SiN_x FILMS ON III-V SEMICONDUCTORS
PRODUCED BY REMOTE PLASMA ENHANCED CHEMICAL VAPOUR DEPOSITION

R. N. S. Sodhi
University of Western Ontario, London, Canada

A remote plasma enhanced chemical vapour deposition reactor attached to an XPS system will be described. This system allows thin films ($<50 \text{ \AA}$) of silicon nitride to be deposited on well characterized semiconductor surfaces which can then be transferred in-vacuo for subsequent XPS analysis. Results from the application of silicon nitride films on clean and oxidized InP surfaces will be presented.

MASK DEPENDENT PLASMA ETCH RATES

T. H. Fedynyshyn
USA

The etch rate of silicon in CF_4/O_2 plasmas is a strong function of the masking material coated on the wafer to define etch patterns. Samples coated with several different metals (silver, copper, chromium, and aluminum) all exhibit a higher silicon etch rate compared to samples coated with a photoresist mask. For silver masked samples, the silicon etch rate can be enhanced by as much as a factor of five relative to a photoresist mask. These results can be explained in terms of a catalytic reaction occurring on the mask surface which gives rise to a local enhancement of the fluorine radical concentration, leading to an increased etch rate of silicon. Evidence for increased fluorine radical concentrations with different metals has been obtained. The dependence of the plasma etch rate on the mask indicates that the nature of the masking material must be taken into account as a variable in plasma etching.

REACTIVE-ION ETCHED GRINSCH LASERS FOR A GaAs MONOLITHICALLY INTEGRATED OPTOELECTRONIC TRANSMITTER

K. E. Fox, C. M. Maritan, M. N. Svilans
BNR, Ottawa, Ontario, Canada, K1Y 4H7

We have recently fabricated a fully integrated monolithic optoelectronic transmitter on GaAs⁽¹⁾. The transmitter incorporated a graded index separate confinement heterostructure GaAsAlGaAs laser diode and monitor photodiode, combined with a GaAs MESFET driver circuit.

This talk emphasizes the use of a reactive-ion etching to form the laser cavity and to electrically separate the transmitter components. A BCl₃/Cl₂ based chemistry was used to produce smooth vertical facets, and eliminated problems often encountered with water vapour in non-load-locked systems.

The dry etch approach to facet formation increases the flexibility in circuit design, but also significantly impacts process flow. Success in integrating the processing for FET devices with the significantly different requirements for optoelectronic components was demonstrated by the production of fully operational transmitters with significant yield.

(1) 'Monolithically Integrated Optoelectronic Transmitter on GaAs', M. N. Svilans, K. Fox, T. Lester, P. Mandeville, C. Maritan, H. Postolek, F. R. Shepherd, A. J. SpringThrope, R. W. Streater, H. W. Willemsen, accepted for the Fourth Canadian Semiconductor Technology Conference, Ottawa, Canada, August 9-11, 1988

INTERACTION OF PLUTONIUM WITH CHEMICAL PLASMAS

Joseph C. Martz

The element plutonium is an active metal which readily forms corrosion products when exposed to most environments encountered in glove box atmospheres. Even when great care is taken to expose the Pu surface only to dry, inert-gas environments, native oxides can be seen to form after a period of several days. Considerable effort has been expended in attempting to find a passivating agent for the Pu surface, generally without success. At present, no suitable method has been found to passivate the Pu surface. In addition, the alpha-emission of the Pu surface leads to rapid, radiation-enhanced polymerization of almost all organic solvents and oils exposed to plutonium. The resulting organic films present numerous problems when further depositions are attempted on the Pu surface. Again, no suitable method has been found to remove these films.

Though plutonium has been widely studied, solution to the above problems remain to be found. However, recent advances in the plasma processing of materials suggest an alternative route to the preservation, passivation, and restoration of the Pu surface. To the best of our knowledge, no investigator has studied the interaction between plutonium and any type of an RF glow discharge. Chemical plasmas (RF glow discharges) are unique environments consisting of energetic charged particles (ion and electrons), neutrals (molecules and radicals), and EM radiation (photons). The degree of ionization is small ($<10^{-3}\%$), but the degree of dissociation in a diatomic plasma can be quite large ($\sim 60\%$ for chlorine). It is these radicals which define the unique chemical environment of the plasma. These species are generally activated toward reaction with a wide variety of surfaces. Surface bombardment by ions also plays a significant role in plasma surface reactions. This bombardment can lead to the sputtering of surface species or to the activation of surface sites for sub-

sequent reaction.

The goal of this research is to examine the interaction of Plutonium with two general classes of chemical plasmas. The growth of stable inorganic Pu compounds, notably Pu oxides, will be studied in various oxidizing plasmas. There is some evidence to indicate that a uniform dioxide might passivate the surface toward further reaction. An oxygen plasma can provide dissociated atoms to the surface eliminating the traditional reaction barrier of surface dissociation. Additionally, the growth of a stable monoxide might be possible under certain conditions. This rare compound is known to be a diffusive barrier to oxygen and a getter for water. The conditions under which the monoxide is thermally prepared might be duplicated within a lower temperature oxygen plasma.

The use of various halogen-containing plasmas to clean the Pu surface will also be studied. The ability to etch organic films from the Pu surface will be examined in both oxygen and water plasmas. Additionally, the actual etching of plutonium itself will be examined. Certain plasma environments might be tailored to selectively etch the oxide in a procedure to restore a previously degraded surface.

To implement this project, a unique plasma reactor tailored to both the special requirements for handling plutonium as well as the needs of this research, has been designed and constructed for use at the Los Alamos Plutonium Processing Facility (Figure 1). The reactor contains two 5-in. electrodes in parallel. Both electrodes are electrically isolated from the main chamber. Carrier and reactive gases are introduced through the center of the powered electrode at a maximum rate of 220 sccm. The chamber is pumped through a port below the lower electrode at a maximum rate of 100 cfm. A computer-controlled throttling valve provides independent feedback control for chamber pressure (Figure 2). Both electrodes are connected to a close-looped cooling system. Provisions for such in situ diagnostics as a quartz crystal microbalance, emission spectroscopy,

mass spectroscopy, and fluoroptic thermometry have been made. The entire experiment is controlled from an IBM PC-AT running ASYST software.

Plasma oxidation of plutonium will be studied to determine the dependence of oxidation rate on plasma power, pressure, flow-rate, oxygen concentration, temperature, and ion-bombardment. The last of these variables is controlled by independently biasing the substrate electrode. In situ oxidation rates will be measured by the microbalance. Chemical composition of the resulting films will be analyzed by Auger and XPS spectroscopy. The latter technique should allow us to determine the oxidation state of the Pu, and hence the monoxide dioxide character of the film. Previous experiments have shown that in order to grow the monoxide, trace quantities of carbon or nitrogen must be present. Addition of these elements can also be controlled during the oxidation.

Etching of Pu will be studied in both chlorine and fluorine containing plasmas. PuF_6 is a gas at room temperature and hence, desorption of etch products from a Pu/F reaction should proceed readily. Plutonium chlorides are not volatile and will probably require substantial ion-bombardment to assist in removal of etch products. Again, these reactions will be studied in a variety of parent gases as functions of power, pressure, flow rate, temperature, and ion-bombardment. The ability to etch the oxide will also be studied. It is desirable to develop a selective chemistry which will remove the oxide in preference to the bare metal. In this respect, the amount of carbon in the parent gas may play a vital role. It has been shown for silicon etching that under certain circumstances, a carbon film is deposited on the surface during the reaction. If an over-abundance of halogen or oxygen is not present, the available etchant species serve only to remove the carbon scale and not to etch the underlying silicon. However, under identical circumstances, the same scale present on a silicon dioxide surface will be volatilized by the oxygen present in the surface itself. This leaves the etchant species available for the remo-

val of the silicon. The result of this is a selective silicon oxide etch. The same chemistry might be used to formulate a plutonium dioxide selective etch.

Lastly, a unique experiment is planned to help elucidate some of the chemical mechanisms occurring during the etch reaction. We plan to provide a modulated bias to the substrate electrode and subsequently observe the appearance of the etch products in the gas phase. By measuring the time-constant for the appearance of the etch products, the kinetic influence of several of the processing parameters upon the concerted reaction may be deduced. Several experimental and theoretical details have been worked out for this procedure. This type of measurement is unique in that it directly measures a kinetic time constant within the actual processing plasma.

ION-SURFACE INTERACTIONS EFFECTS ON THE NUCLEATION AND GROWTH
OF IN FILMS DEPOSITED IN UHV USING LOW ENERGY ION BEAMS

M.-A. Hasan*, J.-E. Sundgren* and J. E. Greene**

*Thin Film group, Department of Physics and Measurements Technology,
Linköping University, 581 83, Linköping, Sweden

**Department of Materials Science, University of Illinois
Urbana, Illinois 61801, USA

A compact, UHV compatible, single grid, low energy ion source was constructed, characterized and used for both primary ion beam deposition of Indium and low energy ion doping during Si Molecular Beam Epitaxy (MBE). The source was used to provide thermal and accelerated In beams with ion to thermal ratios ranging from 0.1 to 60% for ion energies from 100 to 500 eV depending on the ion source operation conditions. For studying ion surface interaction effects during the growth of thin films, a series of thin In films with nominal thicknesses t ranging from 0.3 to 12 nm were deposited under UHV conditions with ion energies ranging from 150 to 400 eV on thin (50 nm) amorphous Si_3N_4 substrates. The as-deposited films were analyzed using transmission electron microscopy (TEM). The films exhibited three dimensional island growth with a bimodal island size distribution. One peak in the distribution shifted to larger islands sizes with increasing coverage, indicating primary island growth and coalescence, while the other remained fixed at low sizes indicating continuous secondary island nucleation. For thermal beam deposition, the overall island density n_{I} increased to a maximum of $\sim 2 \times 10^{12} \text{ cm}^{-2}$ at $t = 0.75 \text{ nm}$ and decreased to $\sim 4 \times 10^{11} \text{ cm}^{-2}$ with increasing t to 10 nm. Ionizing the accelerating the In beam resulted in similar behavior except for considerably lower islands density at high In coverages ($> 0.75 \text{ nm}$). For example, n_{I} was reduced to $\sim 8 \times 10^{10}$ and $\sim 1.2 \times 10^{10} \text{ cm}^{-2}$ for $E_{\text{In}} = 200$ and 300 eV respectively and $t = 10 \text{ nm}$. Size distributions of In islands show a progressive shift towards larger average island

sizes and a decreased secondary island nucleation density as the ion energy was increased. As an example, for $E = 300$ eV there were no islands observed with a diameter, $d < \sim 10$ nm. However, the addition of 1.5 nm of thermally evaporated In to a 10 nm In layer grown with $E_{In} = 300$ eV introduced a secondary nuclei with a size distribution similar to that obtained from a purely thermal layer grown directly on the Si_3N_4 substrate. Reversing the order of the experiment and using an accelerated In beam to deposit additional In decreased the number density of secondary nuclei and resulted in more uniform island size distribution. The results are discussed in terms of ion irradiation induced dissociation and preferential sputtering of small In clusters or nuclei which result in the apparent enhanced adatom surface diffusion.

RECENT MAGNETRON DESIGN IN UNIVERSIDADE DO MINHO CHARACTERIZATION

F. Guimarães, J. B. Almeida, R. Barral*
Universidade do Minho, Laboratório de Física,
P-4719 BRAGA Codex

The Physics Laboratory has been developing planar magnetrons for sputtering research applications for some years. Recently a new design has been tried, characterized mainly by its central magnet and standard 40mm diameter fixing to the chamber, opposed to the previous design, featured with annular magnet and separate water and power supply feedthroughs.

The authors present the results of a comparison between the discharge characteristics of both designs together with the influence of different magnetic fields used in the new design. The plasma characterization has been made with Langmuir probes.

*Instituto Superior de Engenharia do Porto, P-4100 PORTO

WEAR PROPERTIES OF PLASMA NITRIDED NODULAR CAST IRON

M. M. Tosić

The Boris Kidrić Institute of Nuclear Sciences-Vinča
P. O. Box 522, 11001 Belgrade, Yugoslavia

Samples of the pearlitic-sorbite nodular cast iron GGG-80 with the chemical composition: 3.06%C, 2.63%Si, 0.42%Mn, 0.53%Cu have been treated to get a tensile strength of 860 N/mm² (the hardness of 290 HB). The glow discharge plasma nitriding was performed for 9h at about 500°C in the gas mixtures of 80%N₂+20%H₂ and of 55%N₂+45%H₂. The nitriding process parameters were a discharge voltage of 450-600 VDC, a current density of 3-4 mA/cm², and vacuum pressure of 2-4 mbar. Before plasma nitriding, the specimens were hydrogen sputter etched for (1/2)h. Evaluation of nitrided surfaces was conducted by X-ray diffractometry, optical microscopy, microhardness measurements and wear testing.

Two different surface compound layers γ' (corresponding to gas mixture with 55%N₂) and $\gamma'+\epsilon$ (corresponding to gas mixture with 80%N₂) were identified. The surface hardness was about 750HV_{0.3} independent of the phases present. The microhardness profiles reveal diffusion zones extending over a region of 180 μ m. By optical microscopy it has been shown that the thickness of the layer was 6 μ m independent of compound layer type.

The nitrided samples have been tested using the Amsler wear machine, and the results were compared with those for untreated and gas nitrided samples. The latter was performed for 25h at 510°C in the NH₃ gas revealing the surface hardness of about 800HV_{0.3} and having "white layer" of 10 μ m at the top of a diffusion layer of 0.3 mm. The average steady state wear rates were estimated after total of 100,000 cycles, but mass losses were measured after each 5-20,000 cycles. The results have shown that nitriding is very efficient for wear properties improvements. The best results have been obtained with the samples

having γ' phase. Their wear rate is about $50 \times 10^{-15} \text{ m}^3/\text{m}$ to be compared with $65 \times 10^{-15} \text{ m}^3/\text{m}$ for the samples having compound layer of $\gamma' + \epsilon$ mixture (mainly, ϵ). The wear rate of gas nitrided samples, although more than 2 times higher than that for best plasma nitrided samples, is 5 times lower than that of not nitrided ones. By optical microscopy of the surfaces of worn samples, it has been shown that the surface compound layers were present; the worn layers were about $4 \mu\text{m}$ for γ' phase and $5 \mu\text{m}$ for $\epsilon + \gamma'$ case. The microhardness profiles of worn plasma nitrided samples were slightly changed.

The main result of this contribution is that the surface compound layer is responsible for the improvements of the wear properties of plasma nitrided nodular cast iron samples.

A NOVEL MICROWAVE ION BEAM-SYSTEM FOR SURFACE PROCESSING

W. Möhl

Technics Plasma GmbH, D-8011 Kirchheim bei München

K. Fischer

AEG AG S13, Theresienstr. 2, D-7100 Heilbronn

Reactive ion beam etching has become indispensable for fine processing of VLSI devices.

Conventional ion sources of the Kaufman-type suffer from the drawbacks which are introduced by hot filaments and metal electrodes located inside the plasma chamber.

The new developed ion beam system uses a microwave discharge (2.45 GHz) which is self-sustaining and electrodeless. Thus, stable reactive ion beams can be generated for extended periods without the danger of damage to the ion source.

Performance data:

ion outlet diameter	140 mm ϕ
ion acc. voltage	up to 1500 volts
ion curr. density	over 1 mA/ cm ²
pressure range	10 ⁻⁵ - 10 ⁻³ mbar

Extraction grids are made of graphite, plasma chamber is made of quartz.

The new ion beam system will be compared with the conventional ion sources.

Conceptions of appliances and processes of serial production will be discussed as well as the possibilities of collaboration between groups of different countries.

The oral presentation will be given by W. Möhl.

LASER INDUCED FLUORESCENCE MEASUREMENTS IN PLASMA ETCHING PROCESSES

J. P. Booth, G. Hancock, N. Perry and M. J. Toogood
Physical Chemistry Laboratory
Oxford University

Chemical reactions involved in the plasma etching of Si and SiO₂ semiconductor surfaces have been investigated by laser induced fluorescence (LIF) measurements inside a commercial reactive ion etching chamber. CF₄ and mixtures of this with O₂, H₂ and CO₂ have been used as etchant gases, and LIF of CF₂ and CF radicals has been observed as a function of pressure, RF power, gas composition and spatial location with respect to the etched surface. Time resolved measurements using pulsed plasmas have been used to determine loss and production processes at the electrode surfaces. The measurements have been compared with model calculations of the gas phase chemistry using known electron impact production steps and chemical removal processes for the observed radicals.

ADHESION OF THIN FILMS

M. Ozenbas

Dept. of Metallurgical Eng., Middle East Technical University,
Ankara, Turkey

The application of thin films deposited by high-vacuum techniques is an increasingly important aspect of surface coating technology. In all of the applications, the realization of good bonding between the constituent materials determines the performance of the final product. Therefore, great efforts are being directed towards determining the factors which control adhesion as well as establishing new experimental techniques to measure it.

The purpose of this study was to use three different techniques in the determination of the work of adhesion in metal-metal and ABS-metal systems. The techniques used were periodic cracking, microhardness measurements, and the tape test. The results were discussed in terms of the substrate materials, substrate temperature, coating material, and surface roughness of the substrates.

FORMATION OF NICKEL SILICIDES BY ARC-ANNEALING

H. S. Gecim*, Y. Suda**, P. K. John+, B. Y. Tong+ and S. K. Wong++

VLSI Study Group
Centre for Interdisciplinary Studies
The University of Western Ontario
London, Ontario, Canada N6A 3K7

Nickel silicides were formed by arc-annealing. Ni thin films of 1500 Å deposited on Si substrates were annealed by an arc-discharge plasma system. Nickel silicides were observed at the Ni-Si interface. The light pulse of 36 μs duration from the arc plasma and the plasma current were both responsible for the observed silicide formation. Qualitative analysis of silicide layers were carried out by Rutherford Backscattering (RBS) and Auger electron spectroscopy (AES). Scanning Auger microprobe profiling of annealed samples revealed non-uniformity of silicide layers at microscale. Limitations of RBS measurements in examining arc-annealed Ni/Si samples are discussed. Scanning electron micrographs of the samples taken after annealing, with discharge conditions of 7kV and 200 Torr nitrogen, indicated partial segregation of the Ni on the Si surface.

*Permanent address: University of Hacettepe, Electrical and Electronics Engineering Dept., Beytepe, Ankara, Turkey.

**Permanent address: Electrical Engineering Dept., Sasebo Technical College, Sasebo, Japan.

+Department of Physics, U.W.O.

++Department of Chemistry, U.W.O.

EFFECT OF MAGNETIC FIELD AND ITS CONFIGURATION ON THE OUTPUT OF ION OR PLASMA BEAM SOURCES

Ahmet Oztarhan
Dokuz Eylül University, Izmir, Turkey

The basic configuration of hot cathode arc discharge plasma or ion beam sources and their emission mechanism are described. The ion motion in the arc of hot cathode arc discharge plasma or ion source is examined with the assumption that there is a 'Potential Hump' near the anode. It was found that maximum output is determined by the rate of production of ions, which is confined into the volume defined by the magnetic field lines. (magnetic field lines are obtained by computational simulation) on the anode side of the 'potential hump'. Good agreement between the estimated and measured values of the total output supported this view.

Effects of magnetic field and its configuration were studied. The output was maximum when the intermediate electrode and anode pole pieces were saturated.

One can also predict the total output (ions/sec) by knowing the geometry and magnetic field configuration of the ion source together with the desired conditions (arc pressure, arc current).

TECHNOLOGICAL CONSIDERATIONS ON THIN FILM PROCESS BASED ON NTa_2

M. Tudanca, F. Lopez

The setting up of integrated hybrid circuit manufacturing technology for microwave use in telettra española is described.

First, the film growth procedures: reactive sputtering, sputtering, evaporation, and materials used are described. Then follows a discussion of the masking and wet etching techniques. Finally, first results of the technological characterization of the film obtained, are presented.

STUDIES ON MODIFICATION OF SOME FLAMMABILITY CHARACTERISTICS
OF A POLYESTER FABRIC BY PLASMA

G. Akovali and F. Takhrouri
Department of Chemistry and Tumka
Middle East Technical University
Ankara 06531, TURKEY

Plasma treatment of polyester fabric with volatile monomers containing flame retardant elements was made, and the change in some of the flammability characteristics of the plasma treated fabric was studied. The plasma treatment on the fabric caused a decrease in oxygen index values, and a decrease in the burning rates as compared with the untreated polyester fabric. The decrease in (O.I.) values was probably caused by a number of reactions that usually concurrent with plasma polymerization such as crosslinking. It is believed that these reactions had the predominant effect in the changes in flammability behavior of plasma treated fabric, while the concentrations of flame retarding elements on the surface of treated fabric were not enough to show the expected behavior. The results of wettability and TGA tests, as well as SEM data and I.R. and ESR spectra for untreated and plasma treated specimens are included in this work.